Processing Response of Boron Modified Ti-6Al-4V Alloy In (α+β) Working Regime

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Abstract

Titanium alloys like Ti-6Al-4V are the backbone materials for aerospace, energy and chemical industries. Hypoeutectic boron addition to Ti-6Al-4V alloy produces a reduction in as-cast grain size by roughly an order of magnitude resulting in the possibility of avoiding ingot breakdown step and thereby reducing the processing cost. In the present study, ISM processed as-cast boron added Ti-6Al-4V alloy is deformed in $(\alpha+\beta)$ -phase field, where α -lath bending seemed to be the dominating deformation mechanism.

Introduction

Since the introduction of titanium and titanium alloys in the early 1950s, these materials have become backbone materials for the aerospace, energy, and chemical industries. They are very useful light materials that exhibit high specific strength and fracture toughness with a good corrosion resistance for temperatures up to 550–800 K¹. The most widely used titanium alloy is the Ti-6Al-4V (α + β) alloy which is most commonly used in the annealed condition. Microstructure evolution in this alloy was studied with great details by many researchers². ³. The primary problem for any titanium alloy is the large grain size (often in the millimeter range) that evolves during the solidification of the as-cast titanium ingot. Extensive hot working in the β -phase field followed by recrystallization is an effective industrial practice for reducing the grain size of the wrought product. This process, popularly known as 'ingot breakdown', increases the production cost of the finished titanium products and restricts its extensive use to some extent. Any development in terms of grain refinement in as-cast titanium and its alloys is likely to likely to expand the user base of the these materials.

Recently, Zhu et al⁴ have observed that small amount (\sim 0.086 to 0.14 mass%) of boron addition induces a significant refinement of as-cast structure and improvement of mechanical properties like tensile ductility, strength and hardness for cast CP Titanium and Ti-0.5Si alloys. Similar observations are reported by other researchers^{5,6,7}. Tamirisakandala et al⁵ have shown a grain size reduction of as-cast Ti-6Al-4V by about an order of magnitude (from 1700 to 200 μ m) with an addition of only 0.1wt.% boron, while much weaker dependence is observed for boron additions from >0.1% to 1.0%. Wherein Zhu et al⁴ have reported that the tensile ductility of cast Ti-6Al-4V alloy cannot be improved with boron addition, a more recent study by Sen et al⁷ have shown that with the refinement in the microstructure, the yield and ultimate tensile strengths increase whereas the fracture toughness and the threshold for fatigue crack propagation decreases. All these experimental findings leads to the increasing importance of the boron modified Ti-alloys.

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The deformation behavior of normal Ti-6Al-4V alloy is well studied in literature and different deformation mechanisms in both alpha and beta phase fields with various kinds of starting microstructure and composition are already documented by previous researchers $^{8, 9, 10, 11}$. It has been reported that globularization of alpha phase is the deformation mechanism in case of lamellar Ti-6Al-4V during $(\alpha+\beta)$ -processing, wherein fine grained super-plasticity is noticed for equiaxed Ti-6Al-4V in the same working regime. However, any such systematic study on the thermo-mechanical processing of this newly developed boron modified Ti-6Al-4V is still lacking which is the prima facie of the present investigation.

Experimental Procedure

The materials used in this present study are the widely used Ti alloy, Ti–6Al–4V, with 0.1 wt% boron addition (referred to hereafter as Ti64+B). The detailed composition is given in Table 1. The boron was added in the form of elemental boron that completely dissolved in the liquid melt. Compression specimens of 9 mm height and 6 mm diameter were machined from as cast ingots. Isothermal hot compression tests were conducted using a computer-controlled servo-hydraulic testing machine between two Ni-based super-alloy platens. A resistance heating split furnace with SiC elements was used to heat the platens and specimen. The specimens were coated with a borosilicate glass paste for lubrication and environmental protection. The compression test matrix consists of temperatures ranging from 750°–900°C at 50°C intervals and constant true strain rates of 10⁻³, 10⁻¹, 10⁻¹, and 1 S⁻¹. The specimens were deformed to 50% of their original height in each case. The samples were air-cooled to room temperature after deformation. The load-stroke data obtained in compression tests were converted to true stress-true strain curves using the standard procedure. Deformed specimens were sectioned parallel to the compression axis and the cut surface was prepared for metallographic examination. The microstructures of the samples were characterized by scanning electron microscopy¹.

Table-1: Chemical compositions (in wt. %) of boron modified Ti alloy used in the study

Al	V	В	O	Н	C	N	Fe	Ti
6.0	4.0	0.1	0.15	0.005	0.02	0.01	0.13	Balance

Results and Discussions

The microstructure of as cast material is composed of Widmanstäten colonies of alpha lamellae with beta phase being present between alpha lamellae and at the grain boundaries (see Fig 1a). The prior β grain size is ~200 microns with the alpha colonies being finer than 50 microns in. Hexagonal Titanium Boride crystals can be seen at the grain boundary (see Fig. 1b) and forming a typical 'necklace' structure.

Table 2 shows the variation of flow stress with respect to temperature and strain rate for Ti64+B. The flow stress was taken as the stress at a true strain of 0.5. It can be seen that the flow stress increases with strain rate at a fixed temperature and decreases with temperature at a pre-defined strain rate. Fig. 2 show the true stress-true strain flow curves of Ti64+B for various strain rates at a fixed test temperature. At 750°C, stress increases with strain rising up to the maximum value and decreases to a lower value afterwards. At lower strain rate (10⁻³ S⁻¹ and 10⁻² S⁻¹), deformation occurs as a steady state process due to the ease of dynamic recovery during high temperature deformation. However, for higher strain rates

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(10⁻¹ S⁻¹ and 1 S⁻¹), flow stress to cause further strain increases continuously after attaining the peak stress. At 800°C, flow curves are similar to that obtained for 750°C with steady state flow dominating after peak stress for low strain rate (10⁻³ S⁻¹ and 10⁻² S⁻¹). The flow curve of 1 S⁻¹ shows a flow softening after initial peak stress and subsequent increase in flow stress with strain. This kind of flow behavior produces a hump in the flow curve. At 850°C, the flow curves are similar to that obtained at 800°C with steady state flow after peak stress observed even for 10⁻¹ S⁻¹ and 1 S⁻¹ strain rate. The flow curve of 1 S⁻¹ shows usual hump after initial peak stress. At 900°C, the flow curves are similar to that obtained at 850°C with steady state flow after peak stress for almost all the strain rates. The hump in the flow stress can be attributed to dynamic recrystallization of alpha phase 12 which can cause flow softening after the initial peak stress.

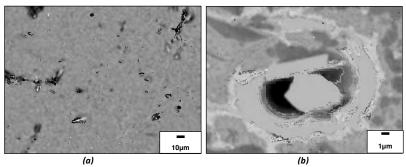


Fig. 1: Microstructure of as-cast Ti64+B sample (a) Widmanstäten alpha colonies, and (b) hexagonal TiB crystals sitting along grain boundaries.

Table 2: Flow Stress at various temperature-strain rate combinations.

		Flow Stress				
Temperature	Strain Rate					
	10 ⁻³ S ⁻¹	10 ⁻² S ⁻¹	10 ⁻¹ S ⁻¹	1 S ⁻¹		
750	189.88	264.94	311	439.56		
800	161.4	161.6	210.63	288.2		
850	95.64	135.65	185.48	240		
900	34.934	85.41	147.14	188		

The temperature and strain rate dependence of flow stress in hot deformation is generally expressed in terms of a kinetic rate equation given by 13 ,

a kinetic rate equation given by
$$\dot{E} = A \sigma^n \exp \left(-\frac{Q}{RT}\right)$$
(1)
e, $\sigma = \text{flow stress}$, $A = \text{frequency factor}$, $Q = \text{apparent active}$

Where $\dot{\mathcal{E}}$ = strain rate, σ = flow stress, A = frequency factor, Q = apparent activation energy, R = Gas constant, T = temperature in Kelvin, and n = stress exponent = 1/m, where m=strain rate sensitivity.

In order to identify the mechanism(s) of hot deformation, the kinetic parameters, n and Q have been evaluated. The variation of flow stresses with strain rates at different temperatures are shown in Fig. 4 on a log-log scale. The slope of the linear fit between the flow stress and strain rate gave the strain rate sensitivity (m) value at that temperature. Table 3 summarizes the strain rate sensitivity of all three materials at each of the working conditions. A good linear fit (regression coefficient not less than 0.98 with error value much less than 0.01) is obtained for most of the working conditions with a very few exceptions. The strain rate sensitivity is quite low (\sim 0.1) at 750°C and 800°C. It increases above 800°C and attains quite high value at 900°C.

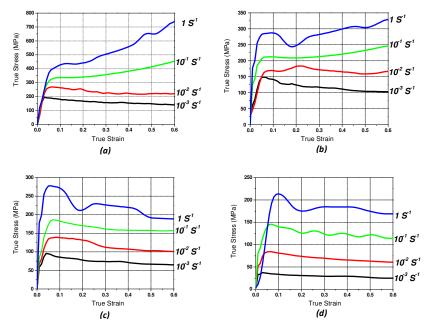


Fig. 2: Flow curves Ti64+B-Axial at various strain rates for the test temperatures of (a) 750° C, (b) 800° C, (c) 850° C, and (d) 900° C.

Similar to strain rate sensitivity, activation energies at different deformation conditions are calculated using the slope of the linear fitting of flow stress vs. inverse of working temperature (see Table 3) in a log-log scale. Satisfactory fitting is obtained for most of the working conditions. The activation energy is particularly high at lower strain rates. It decreases to a lower value as the temperature increase up to 850°C and jumps back to a very high value at 900°C. As reported by previous researchers, in case of α -titanium, activation energy estimated for lattice diffusion is 150 kJ/mole¹⁴, wherein apparent activation energy of 242 kJ/mole was reported for power-law creep¹⁵. These values are relatively low when compared to the range of activation energies that has been obtained in the present

study. However, under certain working conditions, especially at low strain rates like $10^{-3} \, \, \mathrm{S}^{-1}$, the experimental activation energies observed in this study are quite comparable to that reported in literature. Thus at low strain rates and low temperatures, deformation is controlled by dynamic recovery of alpha phase.

It should be noted that the presence of substantial amount of beta (~10 wt%) could be responsible a change the deformation mechanism and hence the activation energy in case of $(\alpha+\beta)$ alloys like Ti-6Al-4V. Comparing the present study with typical strain rate sensitivity (~0.24) and activation energy (~450 kJ/mole) values obtained by earlier researchers for Ti64 using the constitutive equation described above^{8, 9, 10, 11}, slightly higher activation energies are observed in the present case which may be attributed to the presence of additional titanium-boride phases. These can enforce additional barrier to dislocation motion. The strain rate sensitivity values at higher temperature, however, are quite comparable to that already reported.

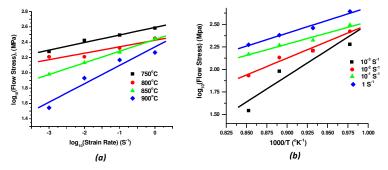


Fig. 3: (a) log_{10} (Flow Stress) vs. log_{10} (Strain rate) plots, and (b) Activation Energy determination from log_{10} (Flow Stress) vs. 1000/T plots.

Table 3: Strain rate sensitivity and activation energy values at various temperatures.

Temperature	Strain Rate Sensitivity (m)	Activation Energy (Q), KJ/Mole
750	0.09869	560.96
800	0.0844	655.94
850	0.15223	363.67
900	0.24035	230.34

Fig. 3 shows the microstructure of the compressed samples at a fixed strain rate of 1 S⁻¹ for different temperatures. The microstructures observed for other strain rates are similar to that of 1 S⁻¹ at all temperatures. It is clearly visible in the micrographs that at all temperatures, alpha lath bending and kinking is the primary deformation mechanism. Individual alpha laths can be considered as single crystals so that under compressive stress, slip through the slip systems causes the laths to rotate and form kinks. Therefore, the colonies which have a favorable orientation with the stress axis, shows different amount of bending in different regions. Another interesting observation is cavitation around titanium boride particles sitting mostly at grain boundaries. These boride compounds, being hard and brittle particles, do not deform at the same rate as does the ductile metal matrix. Therefore a strain mismatch

occurs around the particles which may lead to a cavity formation. However, any other kind of instability (e.g. cavitations at triple junctions, wedge cracking at grain boundaries, adiabatic shear banding etc.) cannot be found even in the regions of secondary tensile stress (see Fig. 4). This seems to be favorable in terms of suitability of this material for thermo-mechanical processing and component manufacturing. From the microstructures, it is difficult to figure out any trace of dynamic recrystallization, although it can be hypothesized that the strain field around the hard boride particles can act as potential sites for nucleation during recrystallization.

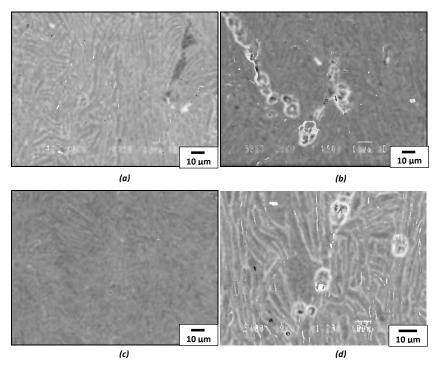


Fig. 4: SEM micrographs of hot compressed Ti64+B samples at a fixed strain rate of $1 \, \text{S}^{-1}$ for different working temperatures of (a) 750°C , (b) 800°C , (c) 850°C , and (d) 900°C . All the micrographs were taken from the mid thickness region where a plane strain condition persists.

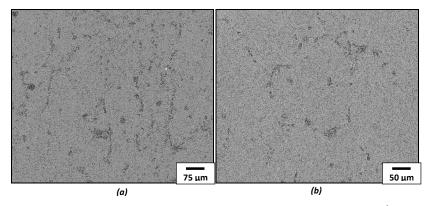


Fig. 4: SEM micrographs of hot compressed Ti64+B samples at a fixed strain rate of 1 S^{-1} for different working temperatures of (a) 800°C, and (b) 900°C. All the micrographs were taken from the side region where a secondary tensile stess is acting.

Conclusion

In the present study, deformation of boron modified Ti-6Al-4V alloy has been carried out in the limited temperature range of 750° - 900° C and strain rate of 10^{-3} to 1 S^{-1} . On the basis of the flow curves and microstructural analysis, the following conclusions have been drawn:

- 1. Steady state flow and substantial flow softening for low strain rates at all temperatures and for high strain rates at high temperatures.
- 2. Kinetic analysis shows the possibility of dynamic recovery in the alpha phase during low strain rate deformation as well as dynamic recrystallization at higher strain rate and temperature.
- 3. Deformation causes the α -lath bending and kinking to accommodate the strain.
- 4. Cavity can be formed around the boride particles due to strain mismatch during deformation.
- 5. No sign of instability can be seen during the deformation process for Ti64+B, possibly due to the ease of nucleation at boride particles during dynamic recrystallization.

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References

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- ⁷ I. Sen, S. Tamirisakandala, D. B. Miracle, and U. Ramamurty, "Microstructural effects on the mechanical behavior of B-modified Ti-6Al-4V alloys," Acta Materialia, 55, 4983-4993 (2007)
- T. Seshacharyulu, S.C. Medeiros, J.T. Morgan, J.C. Malas, W.G. Frazier, and Y.V.R.K. Prasad, "Hot deformation mechanism in ELI grade Ti-6Al-4V", Scripta Materialia, 41(3), 283 (1999)
 T. Seshacharyulu , S.C. Medeiros, W.G. Frazier, and Y.V.R.K. Prasad, "Microstructural mechanisms
- T. Seshacharyulu , S.C. Medeiros, W.G. Frazier, and Y.V.R.K. Prasad, "Microstructural mechanisms during hot working of commercial grade Ti–6Al–4V with lamellar starting structure", Mat. Sc. Engg. A 325, 112 (2002)
 T. Seshacharyulu, S.C. Medeiros, W.G. Frazier, and Y.V.R.K. Prasad, "Hot working of commercial
- ¹⁰ T. Seshacharyulu, S.C. Medeiros, W.G. Frazier, and Y.V.R.K. Prasad, "Hot working of commercial Ti–6Al–4V with an equiaxed α-β microstructure: materials modeling considerations", Mat. Sc. Engg. A 284, 184 (2000)
- ¹¹ S.L. Semiatin, V. Seetharaman, I. Weiss, "Flow behavior and globularization kinetics during hot working of Ti–6Al–4V with a colony alpha microstructure", Mat. Sc. Engg. A 263 (1999) 257–271
- ¹² B. Verlinden, J. Driver, I. Samajdar, and R. D. Doherty, "Thermo-Mechanical Processing of Metallic Materials", Elesevier Ltd., 2007
- ¹³ G. E. Diieter, and D. Bacon, "Mechanical Metallurgy", McGraw-Hill Book Co., 1998
- 14 F. Dyment and C.M. Libanati, "Self-diffusion of $\widetilde{\text{Ti}}$, Zr, and Hf in their hcp phases, and diffusion of Nb 95 in hcp Zr", J. Mater. Sci. 3, 349 (1968)
- 15 M. Doner and H. Conrad, "Deformation mechanisms in commercial Ti (0.5 at. pct) at intermediate and high temperatures (0.3 0.6 T_m)", Metall. Trans. 4, 2809 (1973)

¹ ASM Metals Handbook Volume 2, "Properties and selection of nonferrous alloys and special purpose materials", ASM International, Tenth Edition

iv P. A. Kobryn, S. L. Semiatin, "Microstructure and texture evolution during solidification processing of Ti-6Al-4V," *Journal of Materials Processing Technology*, 135, 330–339 (2003)

³ A. N. Kalinyuk, N. P. Trigu, V. N. Zamkov, O. M. Ivasishin, P. E. Markovsky, R. V. Teliovich, and S. L. Semiatin, "Microstructure, texture, and mechanical properties of electron-beam melted Ti-6Al-4V," Materials Science and Engineering A, 346, 178-188 (2003)

⁴ J. Zhu, A. Kamiya, T. Yamada, W. Shi, and K. Naganuma, "Influence of boron addition on microstructure and mechanical properties of dental cast titanium alloys," Materials Science and Engineering A, 339, 53-62 (2003)

⁵ S. Tamirisakandala, R. B. Bhat, J. S. Tiley, and D. B. Miracle, "Grain refinement of cast titanium alloys via trace boron addition," Scripta Materialia, 53, 1421–1426(2005)

⁶ S. Tamirisakandala, R. B. Bhat, D. B. Miracle, S. Boddapati, R. Bordia, R. Vanover, and V. K. Vasudevan, "Effect of boron on the beta transus of Ti–6Al–4V alloy," Scripta Materialia, 53, 217–222 (2005)